CHAPTER III

EXPERIMENTAL PROCEDURES

Scope

1. Undegummed waste silk was blended with bicomponent fibers which composition was varied in contents, i.e., 20%, 25%, 30% and 35% respectively. The roller carding machine was used for web formation and then areal density was compared between 40 g/m^2 and 60 g/m^2 by initial weighing.

2. Physical properties and mechanical properties were tested by follow JIS L 1085 and JIS L 1096 :

   2.1 Thickness and actual areal density

   2.2 Moisture regain

   2.3 Air permeability

   2.4 Tensile strength, elongation percentage and initial modulus

   2.5 Tear strength

3. Microscopic examination.

Materials

1. Waste Silk

Keba or silk wadding was used as carrier fibers in producing a nonwoven fabric. It is the loose silk mesh around the cocoon which have to be brushed away before reeling, and it can not be used for spun yarns or silk yarns. It was supplied by Chul Thai Agro-industries Co., Ltd, Petchaboon, Thailand which cultivated the Bombyx mori silk. The waste silk was fluff, soft, yellowish white in color, and often collected dirt and burrs, with average length of 29.86 cm (Wongwiboonporn et al., 1992).

2. Bicomponent Thermoplastic Fibers

Thermoplastic fibers were used as binder fibers in producing a nonwoven fabric by thermalbonding technique. Two types of bicomponent thermoplastic fibers were applied.

2.1 Bicomponent Polyolefin Fibers. These bicomponent fibers consist of a polypropylene core and a polyethylene sheath (PE/PP). Their specifications were given as the followings:

Source	:	Chisso Corp., Osaka, Japan.
Trade name	:	ES-CSDL
Appearance	:	White, fluffy and soft, with curl.

Properties (Chisso Corp., 1992) :

Melting point of the sheath component	130 °C
Denier	2.0
Average length	51 mm

2.2 Bicomponent Polyester Fibers. These bicomponent fibers consist of a poly (ethyleneterephthalate) core and a modified poly (ethyleneterephthalate) sheath (mod. PET/PET). Their specifications were given as the followings:

Source : Japan Vilene Co., Ltd., Ibaraki, Japan.

Trade name : T-3380

Appearance : White, fluffy and soft, with curl.

Properties (Japan Vilene Co., Ltd, 1994) :

Melting point of the sheath component	130 °C
Denier	4.0
Average length	51 mm

Preparation of Fibers

1. Degumming of Waste Silk

Some waste silk was degummed to remove the sericin, oil and fats, stain and so on by soda ash, soap and surface active agent. The Over Mieyer Boiler at Shinano Kenshi (Thailand) Co., Ltd. was used for degumming.

1.1 Chemicals Used for Degumming.

Soda ash	3%	o.w.f.
Marseilles soap	4%	o.w.f.
Hydrosulfite	1%	o.w.f.

o.w.f. = on weight fibers

1.2 Procedures. Waste silk was weighed in dry state, and then boiled in aqueous solution (approximately 50:1 in liquor ratio) of soda ash, soap and hydrosulfite for 3 hours in the Over Mieyer Boiler as shown in Figure 3.1. After the degummed silk was washed with water several times, and then dried. The degummed loss (%) was calculated by the following formula,

$$\text{Degumming loss (\%)} = \frac{W - W'}{W} \times 100$$

where, W = mass before degumming

W' = dry mass after degumming

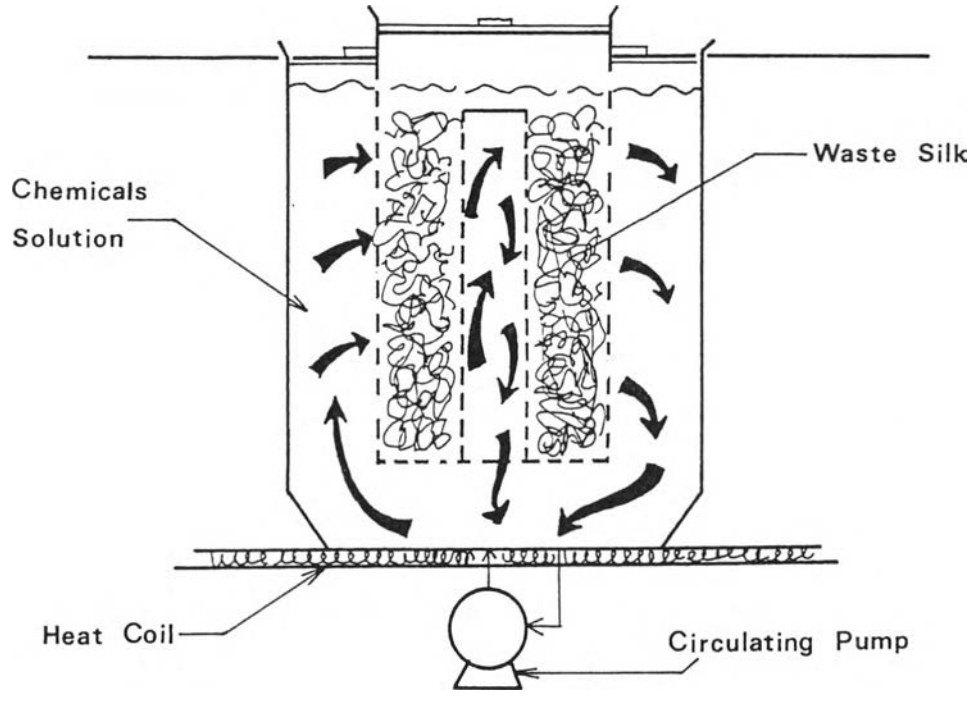

Figure 3.1 Schematic diagram of the degumming process by the Over Mieyer Boiler.

2. Blending

Before blending, waste silk was cleaned in order to remove trash, such as dirt, burrs and remained cocoon or dried silkworms by picking out as much as possible.

By the trial run, it was found that it was difficult to obtain the uniform web which had the areal density lower than 60 g/m^2 by using the available carding machine, because it was encountered the processing problem and it was difficult to control the areal density. The 40 g/m^2 of areal density, however, was also tried to produce to compare with that of 60 g/m^2. At the same time, it was also observed the nonwoven blended with polyolefin binder fibers (PE/PP) gave stiffer fabric hand than that blended with polyester binder fibers (mod. PET/PET). Thus the blended fibers was aimed at containing polyester fibers as binders. The content of binder fibers were varied to 20%, 25%, 30% and 35% at 60 g/m^2 in order to study the correlation between physical properties and the binder fiber contents.

Undegummed waste silk and bicomponent binder fibers were weighed to the required areal density and the binder fiber content on weight given in Table 3.1. Before blending, It was necessary to loosen hard lumps of waste silk and disentangle them by hand. Then waste silk and binder fibers were moderately mixed together by gathering and pulling the mass of both fibers with fingers several times. After the adequate uniformity of blending was obtained, the fiber mass was ready for web formation.

Table 3.1 Blending conditions.

Required Areal Density, g/m^2	Binder Fiber Type	Binder fiber Content , %	Weight , g		Code[a]
			Binder Fiber	Waste Silk	
40	PE/PP	30	12	28	40PE30
60	PE/PP	30	18	42	60PE30
40	mod.PET/PET	30	12	28	40PET30
60	mod.PET/PET	20	12	48	60PET20
60	mod.PET/PET	25	15	45	60PET25
60	mod.PET/PET	30	18	42	60PET30
60	mod.PET/PET	35	21	39	60PET35

(a) Code was representative for conditions used in producing web and was explained in the following ways :

The first numeral group was shown for the required areal density

The second, capital letters group was shown for the bicomponent fibers used as binders.

The last numeral group was shown for the binder fiber content by weight (%)

Web Formation

The laboratory-size roller carding machine 500 Type of Kyowa Machine Co., Ltd., Japan at Japan Vilene Co., Ltd. which is schematically shown in Figure 3.2, was used to produce the web and its specifications were as followings:

Roller width		500	mm
Diameter of the cylinder		450	mm
Diameter of the doffer		450	mm
Delivery speed	max.	15	m/min.
Recommended cylinder speed		150	r.p.m.
Motor		0.4	kW

The web formation was accomplished by the following procedures :

1) The roller carding machine was warmed up for a minute. The cylinder speed was set at 150 r.p.m.

2) The take-in roller was covered with the grease paper to protect the static eletricity building caused by the friction between silk web and metal surface.

3) The fiber mass was throughly sprayed with water to relieve the static, and by this way, water was acted as antistatic agent.

4) The fiber mas was then spread along the feed-in rollers on the conveyor evenly.

5) The licker in was then started. Fibers were passed through the carding machine which put them into a somewhat parallel lengthwise alignment.

6) The take-in roller was then started for a second after feeding the fiber mass.

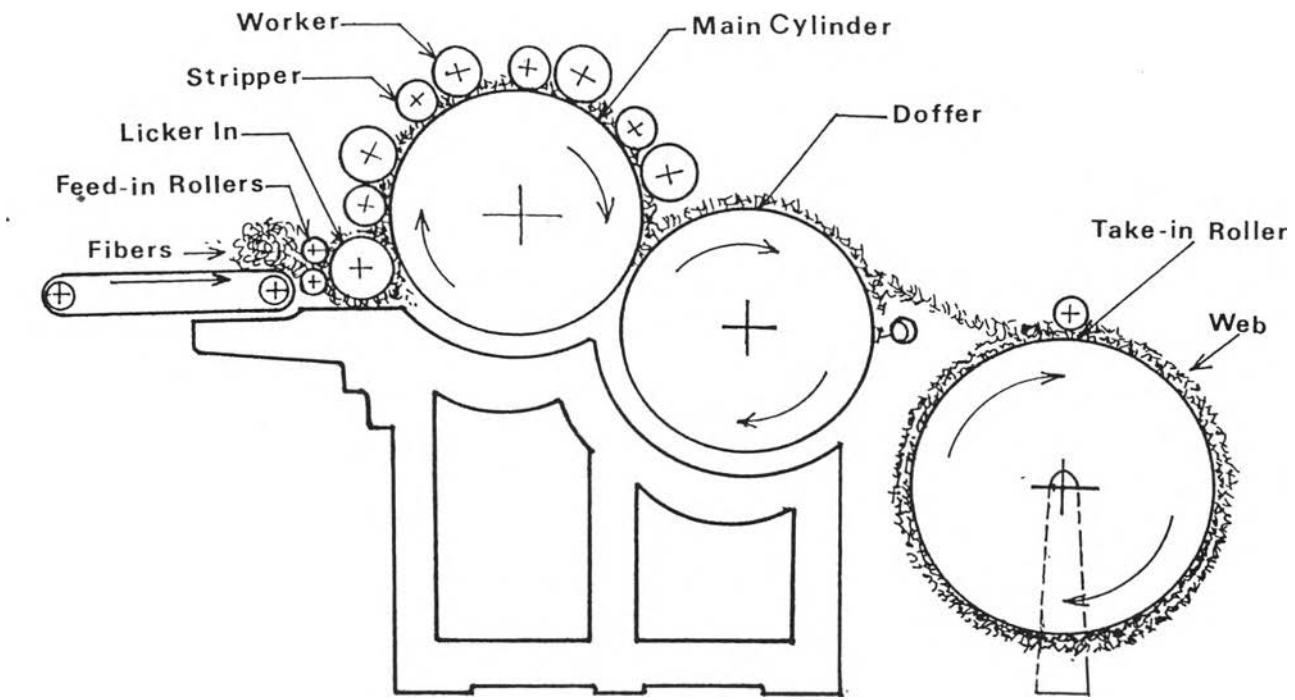

Figure 3.2 Schematic diagram showing formation of web by the roller carding machine.

7) During carding, the thin layer of web was stick on the doffer. When it appeared in sight, it was taken off the doffer altogether by means of the reciprocating doffer comb in front of the doffer. This reciprocating doffer peeled the web off the doffer by a rapid succession of downward strokes. By manual, the web was slowly pulled from the main doffer to the take-in roller which rolled the web about 3-4 layers or until the fiber mass had no more on the doffer.

8) The stacked web was then cut by inserting a pair of scissors into the engraved strip on the take-in roller surface and then cutting the web along the strip.

9) The finished web was spread on the grease paper. The web was given about 166 cm in length with 60 cm in width.

10) Cleaning was required to remove the remained fibers on the main doffer and other rollers before starting the next carding process.

Thermalbonding Procedures

The Low-temperature Fusing Press, JR-600LTS of Asahi Sen-i Kikai Kogyo Co., Ltd., Tokyo, Japan was used for thermalbonding. It is a belt-calendered thermalbonding machine consist of a heated roll and a rubber belt. The schematic diagram of the machine was shown in Figure 3.3, and its specifications were as followings:

Fusing width	600 mm
Pressure range	0 to 5.0 kg/cm^2
Temperature range	room temperature to 180 $^{\circ}C$
Fusing time	4 sec. - 30 sec.

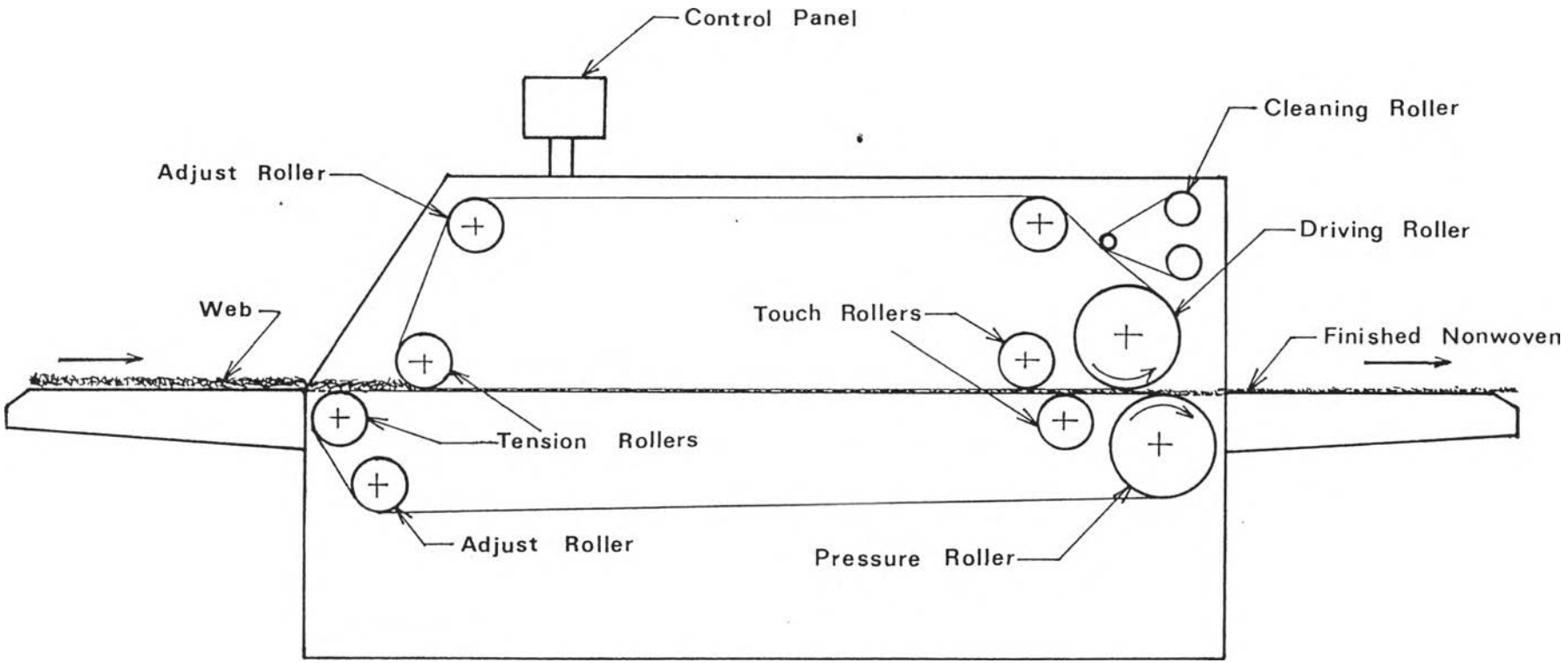

Figure 3.3 Schematic diagram of the belt calendered thermalbonding machine.

Supply air pressure	50 kg/cm^2 (min.)
Supply steam pressure	5 to 7 kg/cm^2
Steam heater steam consumption	6 kg/h (110 °C)
Electric heater capacity	16.2 kW

The prepared webs were sandwiched by two grease papers. The thermalbonding machine was set up for 145 °C and 3.0 kg/cm^2 of pressure. The fusing time was set at 10 sec. When the setting temperature and pressure on the display was steady, the prepared webs were fed into the machine. The webs were heat-bonded by running between the roll and the belt wrapped around the heated roll. Pressure was applied by controlling the tension of the belt against the heated roll and the pressure on the exit guide roll inside the rubber belt. The finished nonwoven came from the machine, was left for cooling, and then the grease papers were peeled off, and the nonwoven fabric was ready for experimental evaluation.

Testing Methods for Nonwoven Fabrics

All specimens for testing mechanical properties were conditioned at the standard condition of test room in accordance with JIS L 1085-1992. The standard condition is standard temperature humidity condition class 2 (temperature 20 $\pm$ 2°C, relative humidity 65 $\pm$ 2%) of JIS Z 8703. However, in case where it was impossible to keep at the above-mentioned conditioned, the temperature and humidity at the time of test was recorded.

1. Thickness

The different five portions of ten specimens of each sample were measured the thickness (mm) by using the micrometer (Mitutoyo) and an average was taken.

2. Areal Density

Relating to each sample, ten sheets of test piece of 20 cm x 20 cm was taken, and respectively weighed the mass (g) under the standard condition. The average value of weight per unit area (g/m^2) was reported.

3. Moisture Regain

Relating to sample, five sheets of test piece of 20 cm x 20 cm were left in a place of test room at standard condition to attain the moisture equilibrium and had become constant weight of the mass before drying. Then the test pieces were kept in a oven drier at a temperature of $105 \pm 2\ ^{\circ}C$ for three hours and were brought to cool down in a desiccator. When they had become constant weight, obtained the absolute dry mass. · The moisture regain (%) was calculated according to the following formula and was expressed by the average value.

$$\text{Moisture regain (\%)} = \frac{W - W'}{W'} \times 100$$

where, W = mass before drying (g)

W' = absolute dry mass (g).

4. Air Permeability

Air permeability testing was determined in accordance with JIS L 1096-1990, Method A using a Frazir type tester, Textile Air Permeability tester, AP-360 D (Daiei Kagakuseiki Seisakusho Ltd., Japan) at Tokyo Metropolitan Textile Research Institute, Tokyo, Japan. The principle of the tester can be explained by using Figure 3.4. When a specimen is attached to one end of the cylinder, the suction fan is adjusted with the rheostat so that the inclined barometer shows a pressure of 1.27 cm on water column. The air volume having passed through the specimen is obtained from the pressure indicated at the time by the vertical barometer and from the type of air hole used by the aid of table attached to this tester.

By using the AP-360 D, five specimens of 30 cm x 30 cm of each sample were prepared. A specimen was clamped to one end of the cylinder, the tester was switched on and then the suction was worked. Testing was accomplished with the auto mode which the air hole diameter was automatically adjusted to obtain the measurable air volume ($cm^3 s^{-1} cm^2$) and expressed it as average of five measurements.

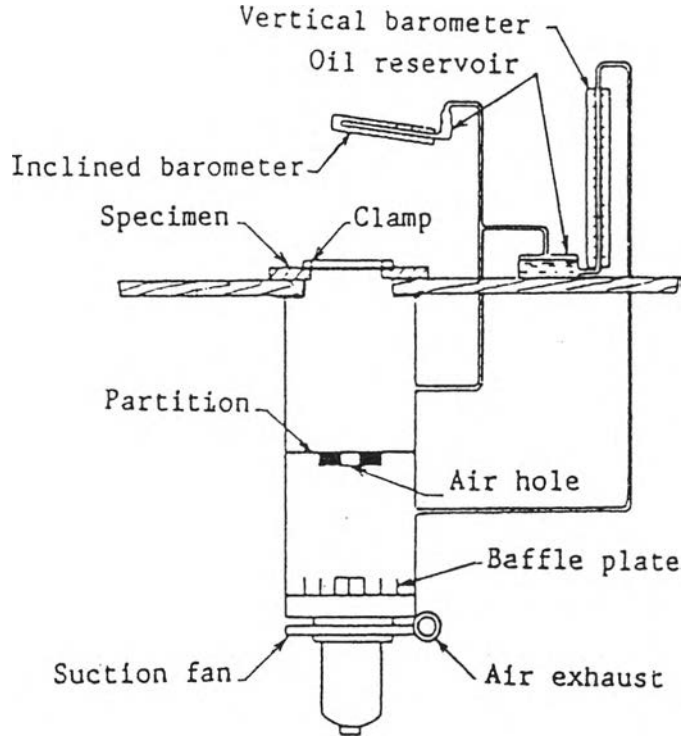

Figure 3.4 Schematic diagram of the Frazir type air permeability Tester (JIS L1096-1990).

5. Tensile Strength and Elongation Percentage

All tensile tests were conducted in accordance with JIS L 1085-1992. Relating to each sample, ten sheets of test piece of 2.5 cm x 20 cm in machine and cross-machine directions, according to JIS L 1096-1990, as appropriate, were prepared. All specimens were left in the test room at standard condition not less than 3 hours. The average thickness of each test piece was obtained by measuring at five positions. The tensile testing machine of constant-rate-of-traverse type was set the grip interval 10 cm, and the tensile speed 30 $\pm$ 2 cm per min., and then the suitable load range was selected such that the break occured between 10-90 % of full scale load. After completely setting, a specimen was clamped with clamping jaws of the machine, and then the machine was operated until the specimen was broken. The stress at break (N/mm^2) and elongation percentage (%) at break was measured. Ten machine and cross-machine direction specimens of each sample were tested and calculated the average value.

The L500 Lloyd universal testing machine fitted with a 150 N load cell and a Lloyd analysis computer software for data acquisition and analysis, were used for testing at Chulalongkorn University. However, the 150 N load cell was too high to test the specimens from cross-maching cuttings resulted in the undetected breaking load. Thus, the Instron 1026 universal testing machine fitted with a 5 kg load cell and a autographic recorder of Textile Industry Division, Department of Industrial Promotion, Ministry of Industry, was used for testing specimens from cross-machine direction cuttings. Figure 3.5 and Figure 3.6 showed the both universal testing machine.

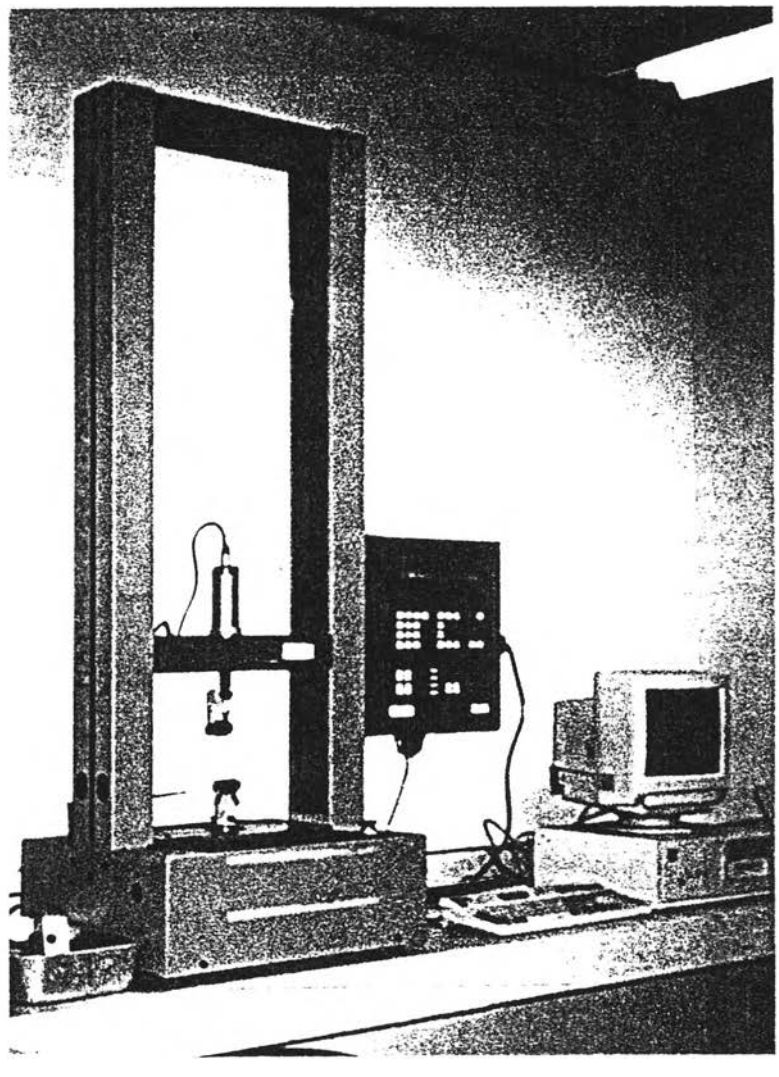

Figure 3.5 The L500 Lloyd universal testing machine.

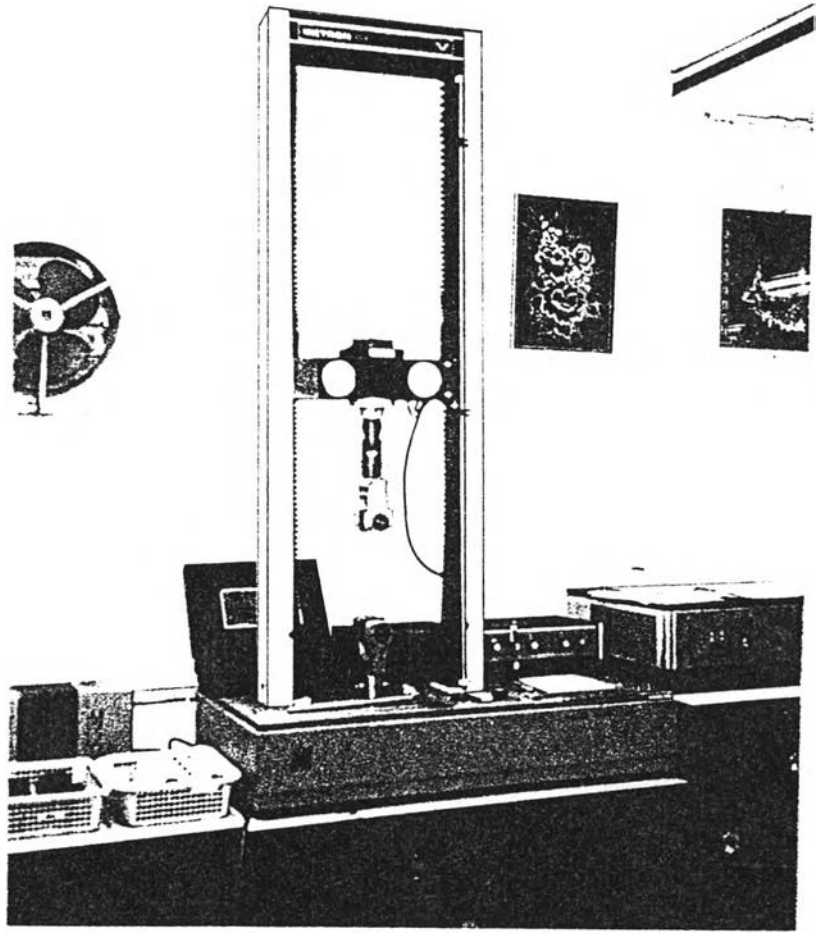

Figure 3.6 The Instron 1026 universal testing machine.

6. Tear Strength

Two methods of tear strength testing, in accordance with JIS 1085-1992, Method A-1 and C were used to compare tear strength of waste silk.

In A-1 method (Single tongue method), the test piece of 5 cm x 25 cm was recommended. However, as mentioned in the objective of this thesis, the waste silk nonwoven was produced to study physical and mechanical properties which can be acceptable for disposable products. According to Atsawahem and Wiraset (1993), the tearing test piece of 5 cm x 20 cm was prepared because of the limitation of samples which cut form the disposable underwears. Thus, in this thesis the test piece of the same size was prepared to obtain the data compared with that of Atsawahem and Wiraset. Ten sheets of each sample in machines and cross-machine directions were taken and cut a streak of 10 cm at the middle of short side at right angles with the side to make two sheets of tongue. The Instron 1026 universal testing machine fitted with 5 kg load cell and a autographic recorder was set the grip interval 10 cm. Each tongue piece of the specimen was placed at right angles with the grip. The maximum load (N) indicating at the time of tearing with the tensile speed of 30 ± 5 cm per min was measured and expressed by respective average values in machine and cross-machine directions.

Another method used was C method (pendulum method). Generally, this method was used in nonwoven industries. Ten sheets of test piece of 6.5 cm x 10 cm were taken respectively in machine and cross-machine directions. By using the Elmendorf tearing tester of Karl Frank, German as shown in Figure 3.7, a streak of

2 cm was cut by a sharp cutting edge at the middle of both grips of test piece at approximate the middle of the long side at right angles with the side. The maximum load (N) indicated when the remaining 4.5 cm is torn, was measured, and an average values of each sample in machine and cross-machine directions were calculated.

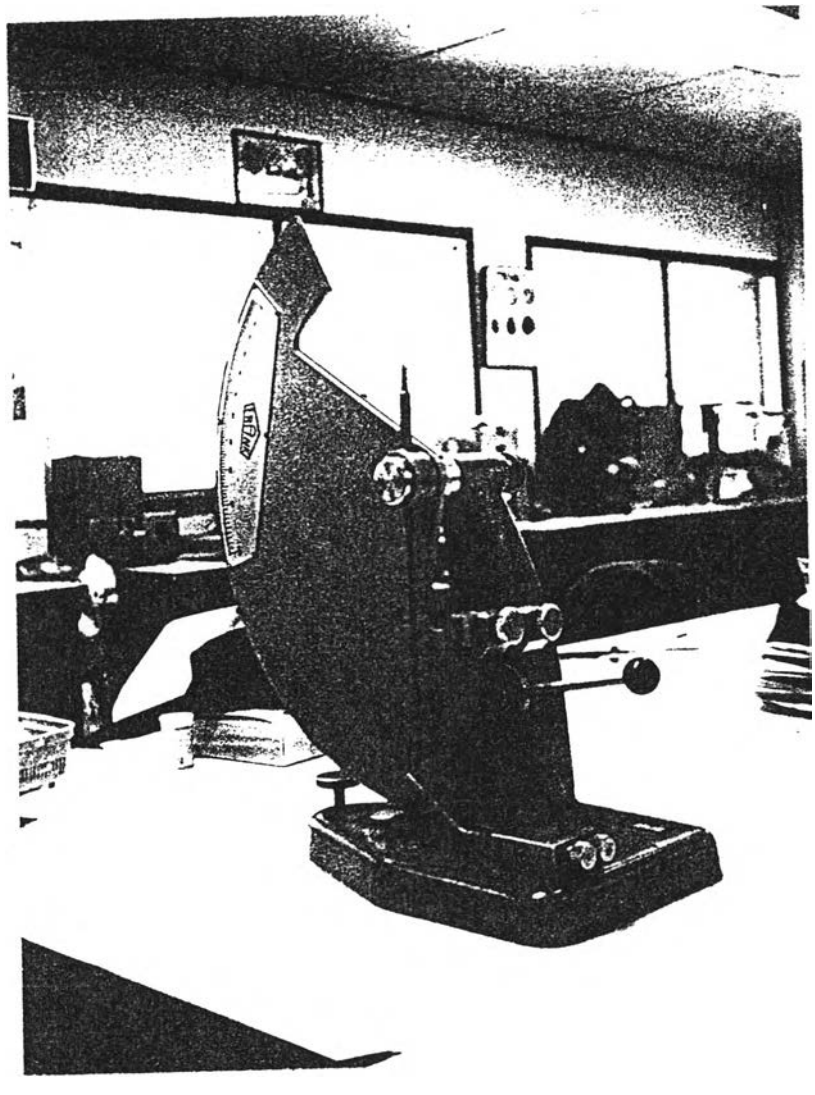

Figure 3.7 The Elmendorf tearing tester.

7. Microscopic Examination

The appearance of nonwoven fabrics was reproduced by the light microscope. More details of the fiber arrangement and the location of bonding points were shown in enlarged views by the scanning electron microscope with an camera attached.

7.1 Optical Microscopy. The Olmpus optical microscope was used to study the alignment of fibers and the texture of nonwoven fabric samples. A test piece was attached to the microscope slide, and then the light was operated to transmit the sample. The suitable magnification was selected to obtain the required enlarged view. The coarse and fine adjustments were required as necessary obtain the clear image of fibers through the scope. The photographs were then taken by an attached camera. Figure 3.8 showed the Olympus optical microscope.

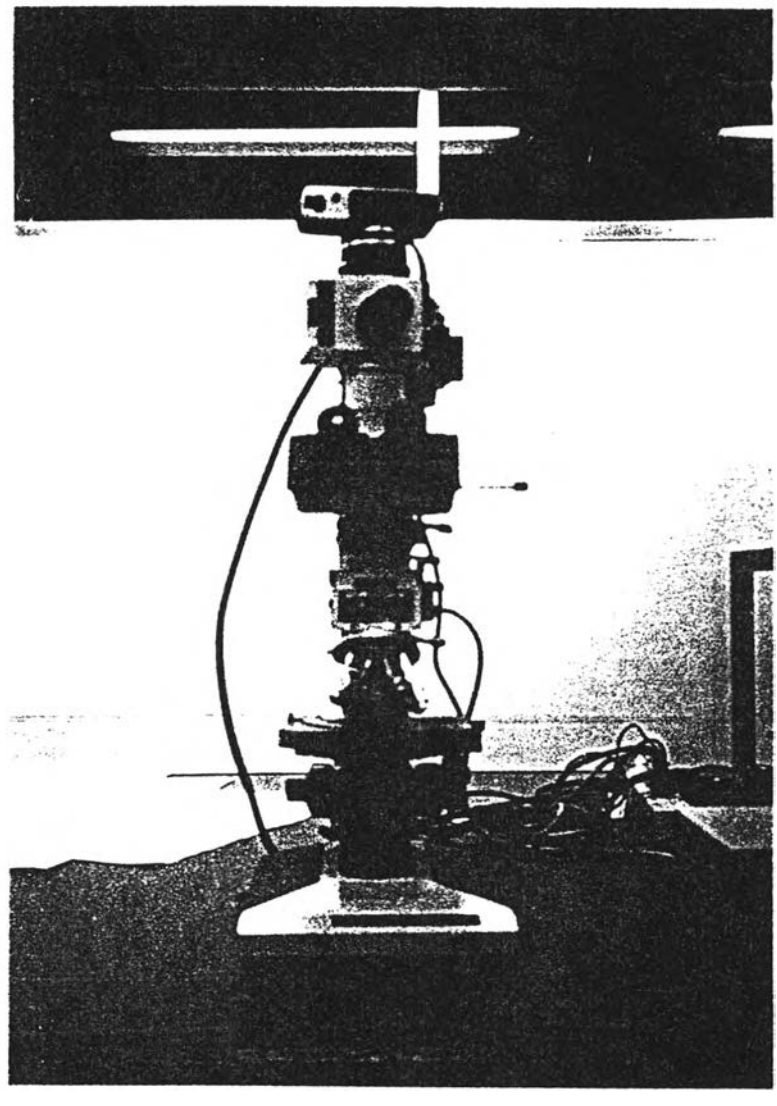

Figure 3.8 The Olympus optical microscope.

7.2 Scanning Electron Microscopy (SEM). In scanning electron microscopy a fine beam of electrons is scanned across the surface of a specimen to which a light conducting film has been applied by evaporation. Secondary electrons, back scattered electrons emitted when the beam hits the specimen are collected to provide a signal used to modulate the intensity of the electron beam in a television tube, scanning in synchronism with the microscope beam. Because the latter maintains its small size over large distances relative to the specimen, the resulting images have great depth of field and a remarkable three-dimensional appearance (Billmeyer, 1984).

Therefore, the pieces of nonwoven samples was sputtered with gold by evaporation and mounted on aluminium stubs. The bonding point examinations was carried out under the JEOL scanning electron microscope and the Hitachi scanning electron microscope.